



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : D21H 21/22, A61K 7/50 // D21H 17:07, 17:20, 17:53, 17:59	A1	(11) International Publication Number: WO 99/06634 (43) International Publication Date: 11 February 1999 (11.02.99)
(21) International Application Number: PCT/IB98/01092 (22) International Filing Date: 17 July 1998 (17.07.98) (30) Priority Data: 197 32 735.4 30 July 1997 (30.07.97) DE (71) Applicant (for all designated States except US): THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): DANNEELS, Allison [BE/BE]; 61, avenue J.B. Depaire, B-1020 Brussels (BE). HILBIG, Klaus [DE/DE]; Hauptstrasse 129, D-65843 Sulzbach (DE). (74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: PROCESS FOR PRODUCING MULTI-LAYERED TISSUE PAPER PRODUCTS		
(57) Abstract A process for producing multi-layered tissue paper products with increased softness comprises the following process steps: production of layered webs of tissue paper, assembly of the layered webs to form a multi-layered web of product which is untreated with respect to softness, and printing of the web of product in the dry state with a softening lotion having the following minimum composition in percent by weight: a quaternary ammonium compound 3 to 30 %, siloxane foam suppressor 0.1 to 3.0 %, aloe vera powder 0.1 to 10 % a polyhydroxy softener, in particular polyethylene glycol 0.05 to 5 % remainder water.		

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Process for producing multi-layered tissue paper products

The invention relates to a process for producing multi-layered tissue paper products, in particular paper handkerchiefs, with increased softness.

With ever-increasing requirements with respect to the quality and comfort of tissue paper products, maximum softness is becoming increasingly important, in addition to improved absorbency and tear resistance - depending on the application of the product. Softness is particularly important in the case of paper handkerchiefs as the human nose is particularly sensitive when sniffing.

It is known from the state of the art, for example EP 0 239 910 A1 or WO 93/09287 A1, to produce a soft absorbent tissue paper by adding to the cellulose fibre-containing pulp a chemical softener solution consisting, for example, of a quaternary ammonium compound such as diethylester dimethyl ammonium chloride and a polyhydroxy softener such as polyethylene glycol in a 1% solution. Other product qualities such as increased resistance to wetting, biological degradability, etc. can be achieved in addition to the improvement in softness, depending on the composition of the active ingredients of this solution.

However, the addition of the softener solution in the wet region of the paper-making machine has drawbacks. For example, the active ingredients are distributed over the entire volume of the layered web of tissue, so a large amount of softener has to be added with respect to the quantity of paper fibre. Furthermore, the positively charged molecules of the quaternary ammonium compound present in the solution have to neutralise the negatively charged fibres in the cellulose pulp. This leads to increased consumption of softener solution. Finally, a quantity of only 0.4 kg remains in the product as active ingredient with an addition of, for example, 11 kg of active softener per tonne of paper pulp. The known process therefore involves great wastage owing to the addition of the softener solution "at the wet end" of the production process.

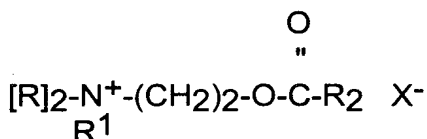
On the basis of the aforementioned problems, the object of the invention is to provide a process for producing multi-layered tissue paper products with increased softness, which is accompanied by more economical consumption of softener substances.

This object is achieved by the process steps mentioned in claim 1, namely:

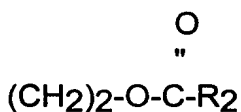
- production of layered webs of tissue paper
- assembly of the layered webs to form a multi-layered web of product which is untreated with respect to softness, and
- printing of the web of product in the dry state with a softening lotion having the following minimum composition in percent by weight:

- a quaternary ammonium compound 3 to 30%
- siloxane foam suppressor 0.1 to 3.0%
- aloe vera powder 0.01 to 10%
- a polyhydroxy softener, in particular polyethylene glycol 0.05 to 5%
- remainder water

preferred quaternary ammonium compound has the formula



and mixtures thereof; wherein each R substituent is a C1-C6 alkyl or hydroxyalkyl group, or mixtures thereof, preferably R is selected from C1-C6 alkyl; R¹ is



or a C13-C19 hydrocarbyl group or mixtures thereof, preferably R¹ is selected from C16-C18 alkyl; R² is a C13-C21 hydrocarbyl group, or mixtures thereof, preferably R² is selected from C13-C17 alkyl; and X⁻ is a compatible anion, preferably chloride or methyl sulfate. As an example, diethylester dimethyl ammonium chloride is to be cited.

The softening lotion is advantageously applied, by the printing of the web of product, virtually only superficially where it imparts pleasant tactile properties to the paper product. The consumption of softening lotion can be halved, for example, in relation to the prior art in that 5.5 kg per tonne of paper are applied. Owing to the printing process, this quantity is found virtually without loss on the end product. Corresponding titration tests on a four-layered paper handkerchief have shown that, of this quantity, 5.3 kg per tonne of paper are found on the two outer layers whereas the softener content is only 0.1 kg per tonne of paper in the two inner layers.

The specified chemical composition of the softening lotion affords various advantages some of which are manifested in conjunction with the printing process. For example, a range of contents of siloxane foam suppressor which is much higher than in the state of the art is used. This foam suppressor is

traditionally used merely to suppress the foam formed on the product by pressure during the packaging process. However, as the packaging stage stresses the product relatively slightly, small proportions of the foam suppressing agent suffice. The proportion of siloxane is typically 0.01% by weight there, that is about 1/10 of the proportion specified in the case of the invention. This high proportion of siloxane counteracts foaming during the printing process which stresses the product much more than the above-mentioned packaging process.

It is also pointed out in this connection that, surprisingly, the softening lotion is insensitive to shearing and so-called droplet penetration is not impaired owing to the great addition of siloxane foam suppressing agent, contrary to expectations. This means that the absorbency and hydrophilic properties of the tissue paper product according to the invention remain virtually just as good in comparison with untreated products.

The advantages of the invention can be summarised as follows:

- Reduced use of softening lotion
- The softening lotion does not leave an unpleasant greasy odour.
- The hydrophilic properties and absorbency of the tissue paper product are not impaired.
- The softening lotion is insensitive to shearing when applied by a printing process.
- The process uses inexpensive chemical substances.

Polydimethyl siloxane, which is mentioned in the state of the art for this purpose, is a preferred foam suppressing agent.

Owing to the acidic adjustment of the softening lotion, it is unnecessary to use preservatives in the tissue paper product. However, intaglio cylinders with a corrosion-resistant surface (for example of ceramic material) are to be used for the printing process.

By the use of a calcium chloride and a soil release agent the viscosity of the emulsion is regulated and a certain storage stability is achieved.

Intaglio cylinders which apply the softening lotion to the external sides of the multi-layered web of product are advantageously used for the printing process. Weights per unit area in the range of 0.5 to 6.0 g/m² on each external side are to be selected for the desired softening effect.

The invention is described in detail hereinafter with reference to an example.

Layered webs of a tissue paper are produced in a quite conventional manner on a paper-making machine to produce a multi-layered paper handkerchief. Further description is unnecessary. It should merely be noted that the weight per unit area of the layered webs is in the range of about 12 to 18 g/m² in each case.

Four such layered webs are assembled in the dry state to form a four-layered untreated web of product, the layers being connected by a stamping process of the type described, for example, in EP 0 755 212 A1. The disclosure of this document is included here.

The dry web of product is finally guided through a pair of intaglio cylinders which applies a softening lotion having a weight per unit area of 1.7 g/m² in each case to the surface of the two external sides of the web of product.

The softening lotion has the following chemical composition:

- diethylester dimethyl ammonium chloride in the form of NN-di (tallowoyl-oxy-ethyl)-NN-dimethyl ammonium chloride with 3 percent by weight
- a siloxane foam suppressor in the form of polydimethyl siloxane with 0.5 percent by weight
- aloe vera powder with 8 percent by weight
- a polyhydroxy softener in the form of polyethylene glycol with 2 percent by weight
- hydrochloric acid with 0.02 percent by weight
- a soil release agent with 0.5 percent by weight
- calcium chloride with 0.2 percent by weight.

The following should be noted with regard to the compounds used:

Further details about the polydimethyl siloxane as foam suppressor and the soil release agent can be obtained from EP 0 239 910 A2 of which the disclosure is included here.

The polyethylene glycol can be obtained as a commercial product known as "PEG 4000" made by BASF, Ludwigshafen, Germany. The same product is sold by the name "PEG 4050" as a 50% solution in water. If the polyhydroxy softener is used in this form, the proportions in the chemical composition of the softening lotion according to the invention should be doubled accordingly.

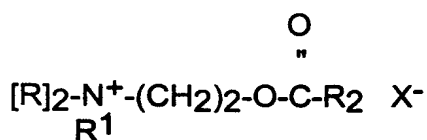
Aloe vera powder is a highly concentrated form of aloe vera of which the concentration is about 200 times that of the liquid obtained by squeezing aloe vera leaves.

Furthermore, the above-mentioned softening lotion in fact per se possesses very good bactericidal properties. This is not only because of the pH-value, but rather in view of the nature of the active itself and the polyhydroxy compound.

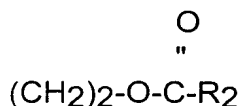
Claims

1. Process for producing multi-layered tissue paper products, in particular paper handkerchiefs, with increased softness, by the following process steps:
 - production of layered webs of tissue paper
 - assembly of the layered webs to form a multi-layered web of product which preferably is untreated with respect to softness, and
 - printing of the web of product in the dry state with a softening lotion having the following minimum composition in percent by weight:

-- a quaternary ammonium compound	3 to 30%
-- siloxane foam suppressor	0.1 to 3.0%
-- aloe vera powder	0.1 to 10%
-- a polyhydroxy softener, in particular polyethylene glycol	0.05 to 5%
-- remainder water.	
2. Process according to claim 1, wherein the quaternary ammonium compound has the formula



and mixtures thereof; wherein each R substituent is a C1-C6 alkyl or hydroxyalkyl group, or mixtures thereof, preferably R is selected from C1-C6 alkyl; R1 is



or a C13-C19 hydrocarbyl group or mixtures thereof, preferably R₁ is selected from C16-C18 alkyl; R₂ is a C13-C21 hydrocarbyl group, or mixtures thereof, preferably R₂ is selected from C13-C17 alkyl; and X⁻ is a compatible anion, preferably chloride or methyl sulfate.

3. Process according to claim 1, wherein the quaternary ammonium compound is a diethylester dimethyl ammonium chloride.
4. Process according to claim 1, wherein the siloxane foam suppressor is a polydimethyl siloxane.
5. Process according to claim 1, wherein the softening lotion is adjusted so as to be acidic, in particular by addition of 0.01 to 0.5% by weight of hydrochloric acid.
6. Process according to claim 1, wherein a soil release agent is added to the softening lotion in a proportion of 0.1 to 1.0% by weight.
7. Process according to claim 1, wherein calcium chloride is added to the softening lotion in a proportion of 0.01 to 1.5% by weight.
8. Process according to claim 1, wherein the softening lotion is applied to the external sides of the web of product by means of intaglio cylinders.
9. Process according to claim 8, wherein the softening lotion is applied to each external side with a weight per unit area of 0.5 to 6.0 g/m².
10. Lotion in particular for use in a process according to claim 1, comprising the following minimum composition in percent by weight:

-- a quaternary ammonium compound	3 to 30%
-- siloxane foam suppressor	0.1 to 3.0%
-- aloe vera powder	0.1 to 10%
-- a polyhydroxy softener, in particular polyethylene glycol	0.05 to 5%
-- remainder water.	

INTERNATIONAL SEARCH REPORT

International Application No

PCT/IB 98/01092

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 D21H21/22 A61K7/50 //D21H17:07,D21H17:20,D21H17:53,
D21H17:59

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 D21H A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 97 06307 A (PROCTER & GAMBLE) 20 February 1997 see page 13, line 1 - line 25 see page 20, line 26 - page 24, line 10 see page 33, line 28 - page 37, line 23 -----	1,4,8,10
A	US 5 624 676 A (MACKEY LARRY N ET AL) 29 April 1997 see claims 1,15,16 -----	1
A	EP 0 239 910 A (PROCTER & GAMBLE ; PROCTER & GAMBLE EUROP (BE)) 7 October 1987 cited in the application see the whole document -----	1-7,10



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

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Date of the actual completion of the international search

6 October 1998

Date of mailing of the international search report

15/10/1998

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/IB 98/01092

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9706307 A	20-02-1997	US 5705164 A AU 6598996 A EP 0842327 A	06-01-1998 05-03-1997 20-05-1998
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PUB-NO: WO009906634A1

**DOCUMENT-
IDENTIFIER:** WO 9906634 A1

TITLE: PROCESS FOR
PRODUCING MULTI-
LAYERED TISSUE PAPER
PRODUCTS

PUBN-DATE: February 11, 1999

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APPL-NO: IB09801092

APPL-DATE: July 17, 1998

PRIORITY-DATA: DE19732735A (July 30, 1997)

INT-CL (IPC): D21H021/22 , A61K007/50

EUR-CL (EPC): D21F011/04 , D21F011/14 ,
D21H021/22

ABSTRACT:

CHG DATE=19990402 STATUS=O>A process for producing multi-layered tissue paper products with increased softness comprises the following process steps: production of layered webs of tissue paper, assembly of the layered webs to form a multi-layered web of product which is untreated with respect to softness, and printing of the web of product in the dry state with a softening lotion having the following minimum composition in percent by weight: a quaternary ammonium compound 3 to 30 %, siloxane foam suppressor 0.1 to 3.0 %, aloe vera powder 0.1 to 10 % a polyhydroxy softener, in particular polyethylene glycol 0.05 to 5 % remainder water.